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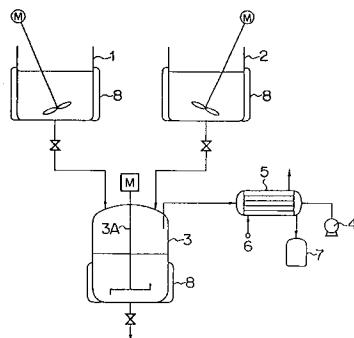
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W-8000 München 80(DE)(54) **Method for preparing a hydrophobic substance for use in photography.**

(57) A hydrophobic substance for photographic use is dispersed in water. A first solution is prepared by the hydrophobic substance and a low boiling point organic solvent which is insoluble in water. A second solution is prepared by water and a surfactant being dissolved in said water. The first solution and the second solution is mixed to form a mixture. Thereafter, the mixture is stirred under a reduced pressure condition to evaporate the organic solvent from the mixture, thereby the hydrophobic substance being dispersed in the water can be obtained.

Fig. 1

BACKGROUND OF THE INVENTION

The present invention relates to a method for preparing a hydrophobic substance for use in photography, and more particularly relates to a method for preparing a hydrophobic substance characterized in that: all kinds of hydrophobic substance can be precipitated when oil-in-water type dispersion is formed only by water-insoluble low boiling point organic solvent substantially without using high boiling point organic solvent; and a hydrophobic substance of a predetermined particle size can be easily obtained.

When a photosensitive material is prepared, a dispersion of a predetermined particle size is obtained in such a manner that: after a hydrophobic substance such as a color-image forming compound (a coupler), compound for use in diffusion transfer material, anti-color fogging agent, anti-color fading agent, anti-color mixing agent, UV absorbent and whitening agent, has been dissolved in a high boiling point organic solvent such as alkylphthalate, phosphoric acid esters, citric acid esters, benzoic acid esters, alkylamides and fatty acid esters, the solution is dispersed in water. In this case, it is very important to control the particle size of the dispersed substance. Especially when a coupler is used to finally prepare a photosensitive material, the particle size has influence on photographic characteristics.

In the field of photosensitive material, the following proposals have been made as a method to obtain oil-in-water type dispersion in which high boiling point organic solvent is dispersed.

(1) In the official gazette of Japanese Patent Publication No. 15005/1984, an embodiment has been disclosed in which a high pressure dispersing apparatus is used and the solution is dispersed by both shearing and colliding force.

(2) In the official gazette of Japanese Patent Application Open to Public Inspection No. 293537/1986, an embodiment has been disclosed in which the solution is dispersed by a high speed mixer.

The following methods has been disclosed as a method by which a hydrophobic substance is precipitated in the form of fine particles.

(3) In British Patent No. 1,193,349 and European Patent Application Open to Public Inspection No. 374, 837, a method has been disclosed in which a hydrophobic substance is precipitated and stable colloid is obtained, wherein the phenomenon is utilized in which the dissolution degree of a hydrophobic substance differs according to pH.

(4) In British Patent, a method has been disclosed in which: after coupler has been dissolved in a low boiling point organic solvent, it is coated; and in the coating process, the aforementioned low boiling point organic solvent is evaporated so that the solvent can be removed.

However, in the case of the aforementioned oil-in-water type dispersion method in which a high boiling point organic solvent is utilized, the high boiling point organic solvent remains in a coating film, so that the thickness of the film is increased. Accordingly, sharpness is deteriorated, and further color bleeding is caused by the high boiling point organic solvent.

Furthermore, the methods disclosed by British Patent No. 1,193,349 and European Patent Application Open to Public Inspection No. 374, 837 are disadvantageous in that: the methods can be applied only to coupler in which the solubility is changed by pH. Furthermore, the methods can not be applied to the coupler having an ester group capable of being hydrolyzed upon the change of pH value. Precipitation caused by the change of pH can be quickly stabilized by a binder or surface active agent, so that a fine particle size can be obtained. However, it is difficult to control the particle size.

On the other hand, in order to simultaneously remove unnecessary organic solvent and salt generated by neutralization, it is necessary to adopt the noodle washing method and ultrafiltration method. However, a long period of time and a large amount of water are required, and further, polluted waste liquid must be processed, so that consideration must be given to the environmental problems.

The method of British Patent 1,099,414 is disadvantageous in that: oil-in-water type dispersion comes into contact with a solvent over a long period of time; and accordingly, the oil-in-water type dispersoid coagulates, so that coarse particles are generated.

SUMMARY OF THE INVENTION

The first object of the present invention is to solve the aforementioned problem by forming a oil-in-water type dispersion without substantially using a high boiling point organic solvent.

The second object of the present invention is to provide a method for preparing a hydrophobic substance for photographic use by which fine particles of a hydrophobic substance can be obtained, irrespective of the kind of the hydrophobic substance, whereby the particle size can be easily controlled.

The aforementioned problems can be solved by adopting a method for dispersing a hydrophobic substance for photographic use in water characterized in that: the first solution in which the aforementioned

hydrophobic substance is substantially dissolved only in a water-insoluble low boiling point organic solvent, and the second solution in which only a surfactant, or both a surfactant and a binder are dissolved in water, are dispersed in each other so as to form a oil-in-water type of dispersion; and when the dispersion is stirred under reduced pressure, the aforementioned water insoluble low boiling point organic solvent
 5 contained in the aforementioned oil-in-water type dispersion, is removed so as to form finally dispersed precipitates of a hydrophobic substance.

When the aforementioned dispersing operation is performed, a high speed stirring type of dispersing apparatus may be utilized so that the dispersion is stirred under the condition that the dispersing circumferential speed is not less than 10 m/sec.

10 It is preferable to start the stirring operation under reduced pressure within 5 minutes after the oil-in-water type of dispersion has been formed.

In the process of removing the aforementioned water-insoluble low boiling point organic solvent, it is preferable to continue the removing operation until the content of the water-insoluble low boiling point organic solvent becomes not more than 1 wt%.

15 According to the present invention, the coating thickness can be prevented from becoming thick, sharpness can be improved, and the occurrence of bleeding of color can be prevented without using a high boiling point organic solvent.

Further, according to the present invention, fine particles of a hydrophobic substance can be obtained and the particle size can be easily controlled irrespective of the kinds of hydrophobic substances.

20 BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a schematic drawing showing an example of the manufacturing apparatus of the present invention; and

25 Fig. 2 is a correlation diagram between the standing time and the average particle size of the obtained hydrophobic substance.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

30 The present invention will be described in detail as follows.

The summary of the invention will be explained referring to Fig. 1 showing a manufacturing apparatus in which a coupler is utilized as a hydrophobic substance.

That is, in the first dissolution tank 1, the first solution is obtained by dissolving a hydrophobic coupler in a water insoluble low boiling point organic solvent. On the other hand, in the second dissolution tank 2, the second solution is obtained by dissolving both a surface active agent and a binder in water. The
 35 obtained first and second solution are emulsionized and dispersed in a dispersion tank 3 provided with a high speed stirring type dispersing apparatus 3A so that an oil-in-water type of dispersion is made. After that, the dissolution tank 3 is evacuated by a vacuum pump 4, and at the same time the high speed stirring type dispersing apparatus 3A is operated so that the water insoluble low boiling point organic solvent is
 40 evaporated. The solvent vapor is condensed by cooling with a refrigerant 6 in a heat exchanger 5 so that the water insoluble low boiling point organic solvent is removed in the form of liquid, which is recovered into a recovery tank 7, and at the same time a coupler is precipitated from the recovered liquid in the dissolution tank 3. Then, a stabilized solid-liquid dispersion in which a solid is dispersed in a liquid medium, can be obtained by the action of a surfactant and a binder.

45 In the aforementioned example, the first dissolution tank 1 and the second dissolution tank 2 may be provided with a heating jacket 8 and a heater in order to facilitate dissolution, dispersion, and stabilization so that the temperature of the solution can be controlled. A dissolver type, oar type, propeller type, and homomixer type of high speed stirring dispersing apparatus can be adopted. It is preferable to maintain the temperature in the dispersion tank 3 at 15 - 60 °C.

50 On the other hand, the first and the second solution may not be put into the dispersion tank 3 separately, but may be mixed with each other and put into the tank 3. Usually, after the first and the second solution have been put into the dispersion tank 3, the operation of the high speed stirring type of dispersing apparatus is started. However, in the case of continuous dispersion, the dispersing operation can be performed while the first and the second solution are being supplied during the operation of the high speed
 55 stirring type of dispersing apparatus 3A.

The operation conducted during dispersion has great influence on the dispersing property. The particle size of the dispersion mainly depends on the rotation speed of the dispersing apparatus, the stirring time, and the amount of the activator. The dispersing circumferential speed is preferably not less than 10 m/sec

and not more than 100 m/sec.

It is preferable to stir under reduced pressure immediately after the oil-in-water dispersion has been made in the process of dispersion. Depending on the standing time from the completion of dispersion to the start of stirring operation, the particle size to be obtained grows as shown in Fig. 2. Consequently, it is desirable to restrict the standing time within 5 minutes. Fig. 2 shows an example in which the solution temperature is maintained at 45° C during the process of standing.

The stirring time relates to the particle size to be obtained. However, it is preferable to maintain the stirring time in 3 - 60 minutes. When an evacuating operation is performed in order to remove the water-insoluble low boiling point organic solvent, the pressure is gently reduced to 100 Torr, and preferably the evacuating operation may be conducted according to the method disclosed in the Japanese Patent Application open to Public Inspection No. 102303/1990. Further, the temperature of the solution in the case of removal of the water impermeable low boiling point organic solvent is preferably 40 - 80° C, and more preferably 50 - 70° C. The removal of the water-insoluble low boiling point organic solvent is preferably conducted until its content becomes not more than 1 wt%.

The amount of the surface active agent to be added relates to the kinds of binder and coupler of the water-insoluble low boiling point organic solvent. However, the amount of the surface active agent is preferably in the range from the critical micelle concentration to 50 times of the critical micelle concentration. When these factors are controlled, the liquid particle size can be controlled to be 0.01 - 3 μ m in the present invention. The circumferential dispersing speed in the process of dispersion and the stirring circumferential speed in the process of removal of the water-insoluble low boiling point organic solvent, may be different from each other.

A solution in which only the surface active agent is dissolved in water may be utilized as the second solution without using the hydrophilic binder. When the hydrophilic binder is used, the content may be 3 - 30 wt% in the solution put in the dispersing tank 3.

In the present invention, the boiling point of the water-insoluble low boiling point organic solvent is not more than 100° C, and more preferably not more than 85° C. The specific examples are ethyl acetate, methyl acetate, m-hexane, m-pentane, benzene, cyclohexane, cyclopentane, chloroform, and dichloromethane.

In the present invention, the objective hydrophobic substance is substantially dissolved only in the water-insoluble low boiling point organic solvent. In this case, it is preferable that a high boiling point organic solvent is not contained at all.

When a high boiling point organic solvent such as dimethyl form-amide (DMF) is used as an auxiliary solvent, a small amount of the solvent, for example, not more than 5 wt% can be added so that the sharpness is not deteriorated and bleeding of dye is prevented.

A coupler which is a hydrophobic substance applied the various photographic materials of the present invention, is a color image forming compound which forms a dye upon reaction with an oxidized product of color developing agent, for example aromatic amine (the first class amine). In general, a non-diffusible coupler having a hydrophobic group referred to as a ballast group, is preferably used, and either 4 equivalent or 2 equivalent coupler may be used. The aforementioned coupler includes a colored coupler provided with color correction effect, and a DIR coupler which releases a developing inhibitor while developing is conducted. A widely known closed ketomethylene coupler can be utilized as a yellow coupler. Benzoyl acetoamide compounds and pivaloyl acetoamide compounds are advantageously used.

Pyrazolone compounds, indazolone compounds, and cyanoacetyl compounds can be used as a magenta coupler, wherein pyrazolone compounds are advantageous.

Phenol compounds and naphthol compounds can be used as a cyan coloring coupler.

A coupler disclosed in the official gazette of Japanese Patent Application Open to Public Inspection No. 42121/1977 can be used as a colored coupler.

A coupler disclosed in the Japanese Patent Application Open to Public Inspection No. 69624/1977 can be used as a DIR coupler. Except for the aforementioned DIR coupler, a compound which releases a developing inhibitor during a developing process may be contained in the photographic material, and for example, a compound disclosed in the official gazette of Japanese Patent Application Open to Public Inspection No. 9116/1978 can be used.

The following non-ionic surface active agents are used for the present invention: a saponin (steroid) derivative; an alkyleneoxide derivative (for example, polyethylene glycol, condensation of polyethylene glycol/polypropylene glycol, polyethylene glycol alkyl or alkylaryl ether, polyethylene glycol ester, polyethylene glycol sorbitan ester, polyalkylene glycol alkylamine or amide, and polyethylene oxide additive product of silicon); a glycidol derivative (for example, alkenyl succinic acid polyglyceride, and alkylphenol polyglyceride); fatty acid ester of polyhydric alcohol; alkyl ester of sugar; and urethane or ether.

The anion surface active agents containing the following acidic groups such as a carboxyl group, a sulfo group, a sulfate ester group, and a phosphoric ester group are used for the present invention: saponin of triterpenoide, alkylcarboxylic acid salt, alkylsulfonic acid salt, alkylbenzene sulfonic acid salt, alkyl-naphthalene sulfonic acid salt, alkylsulfate ester, alkyl phosphoric ester, N-acyl-N-alkyltaurine, sulfosuccinic acid ester, sulfoalkylpolyoxyethylene alkylphenyl ether, and polyoxy ethylene alkyl phosphoric ester.

The following amphoteric surface active agents are used for the present invention: amino acid; aminoalkyl sulfonic acid; aminoalkyl sulphuric acid or phosphoric ester; alkylbetaine; amineimide; and amineoxide.

The following cationic surface active agents can be used for the present invention: heterocyclic quaternary ammonium salt such as alkylamine salt, aromatic or aliphatic quaternary ammonium salt, pyridinium, and idazolium; and aliphatic or heterocyclic phosphonium or sulfonium salt.

Especially, an anionic surface active agent such as dodecyl benzene sulfonic acid, dodecyl sodium sulfate, and "Aerosol A102" (made by Cyanamid Co.) is preferably used.

Protein such as gelatin, a gelatin derivative, a graft polymer of gelatin and other high polymers, albumin, and casein is used for the aforementioned water soluble binder.

A cellulose derivative such as hydroxyethyl cellulose, carboxymethyl cellulose, and cellulose sulfate ester, and a sugar derivative such as sodium alginate and a starch derivative are also used for the aforementioned water soluble binder.

Synthetic hydrophilic high molecular substance such as polyvinyl alcohol, polyvinyl alcohol acetal, poly-N-vinyl pyrrolidone, polyacrylic acid, polymethacrylic acid, polyacrylic amide, polyvinyl imidazol, and polyvinyl pyrazole, or synthetic hydrophilic high molecular substance of a copolymer of the compounds described above, are used for the aforementioned water soluble binder.

Usually, it is desirable to use gelatin for the binder.

A dye developer diffusible dye releasing coupler (DDR coupler) and diffusible dye releasing reducer (DRR compound) are used for the aforementioned compound for diffusion transfer use.

For example, a hydroquinone derivative, aminophenol derivative, gallic acid derivative and ascorbic acid derivative are used as the aforementioned anti-stain agent.

For example, a hydroxybenzene derivative, dihydroxynaphthalene derivative, aminonaphthol derivative, sulfonamide phenol derivative, and sulfonic amide naphthol derivative are used for the aforementioned anti-fading agent.

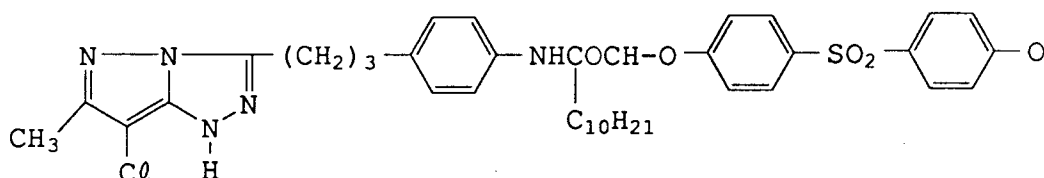
For example, a benzotriazole compound substituted by an aryl group, 4-thiazolidone compound, benzophenone compound, cinnamic acid ester compound, butadiene compound, and benzoxazole compound can be used for the aforementioned ultraviolet ray absorbent. Further, an ultraviolet ray absorbing coupler and ultraviolet ray absorbing polymer may be used.

For example, a compound of stilbene, triazine, oxazole or cumalin is used for the aforementioned whitening agent.

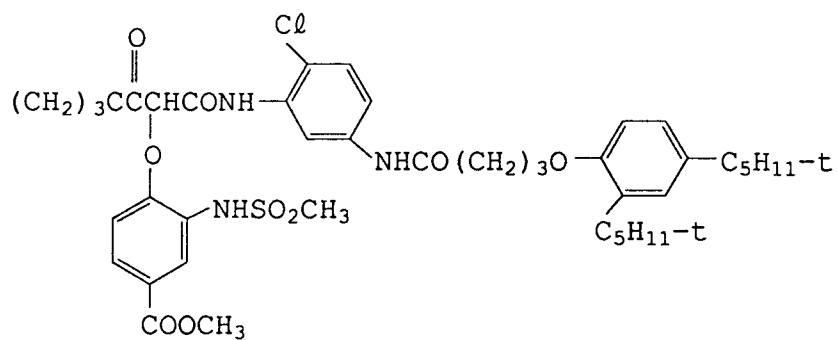
Next, an example will be shown to clarify the effect of the present invention.

The following coupler and surface active agent were used in the example of the present invention.

<Coupler (1)>



<Coupler (2)>



⟨Surface active agent (1)⟩

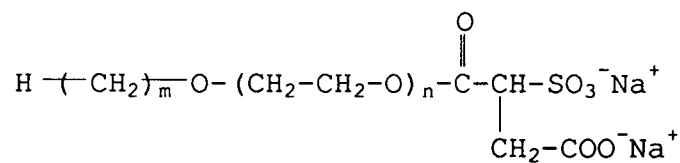
Dodecyl benzene sodium sulfonate

⟨Surface active agent (2)⟩

Dodecyl benzene sodium sulfate

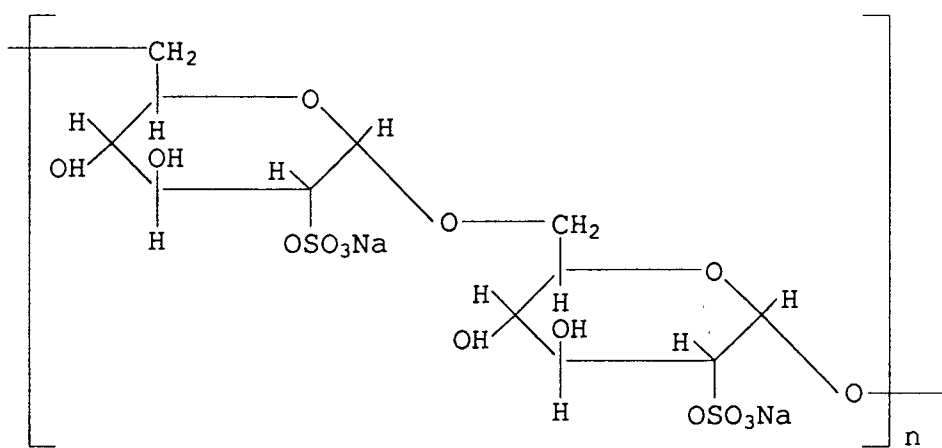
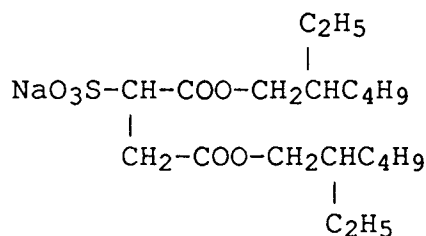
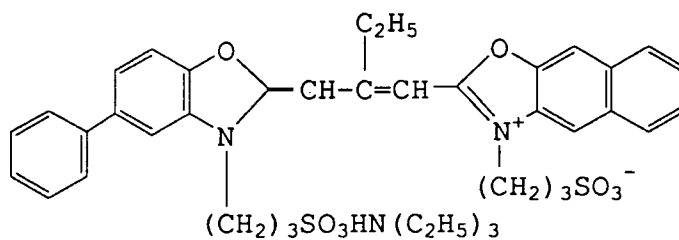
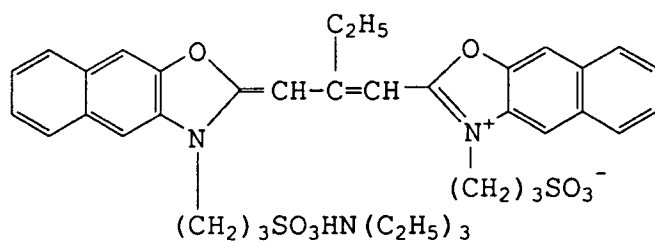
⟨Surface active agent (3)⟩

(Aerosol A102)



$$m=10\sim 12, \quad n=3\sim 5$$

The following were used for spectral sensitizers (S-1), (S-2), a coating aid (Su-1), and a thickening agent (V-1).



EXAMPLE 1

50 The first solution of the following composition was dissolved for one hour at 60° C.

Coupler (1)	5 kg
Ethyl acetate	10 l
DMF	300 ml

55 On the other hand, the second solution of the following composition was dissolved for one hour at 60° C.

Water 50 l

Surface active agent (1)

2.5 l (Concentration 25%)

Polyvinyl pyrrolidone 2.5 kg

Subsequently, the first and the second solution were mixed, and dispersed by a dispersing apparatus provided with a disperser having a diameter of 10 cm, at a circumferential dispersing speed of 20 m/sec for 10 and 20 minutes, and at a circumferential dispersing speed of 30 m/sec for 10 minutes. Then, the solution was evacuated and stirred until the residual concentration of ethylacetate became not more than 0.3 wt% so as to remove ethylacetate, and subsequently, the solution was diluted with water to make 100 l.

The dispersed particle size of thus obtained solid-liquid dispersion in which a solid was dispersed in a liquid medium was measured by means of the photon correlation method, and the results shown in Table 1 were obtained. A photosensitive material was made by the method described later, and its photographic characteristics were investigated to obtain the results shown in Table 1.

In this case, the sharpness was measured by means of the modulation transfer function method. The detailed results are described on pages 430 - 437 of "Photographic Chemistry" (which was written by Akira Sasai and published by Shashin Kogyo Shuppansha). In this measurement, the photosensitive material was exposed to white light through an MTF chart and developed, and its sharpness was represented by MTF values at 40 cycle/mm.

TABLE 1

Circumferential Dispers-ing Speed	Inventive Example			Blank	Comparative Example
	20 m/sec		30 m/sec	25 m/sec	-
Dispers-ing Time	10 min	20 min	10 min	15 min	-
Particle Size	485 nm	125 nm	90 nm	160 nm	50 nm
Dmax	109%	120%	108%	100%	103%
MTF	0.66	0.68	0.70	0.58	0.68

The aforementioned comparative example in Table 1 is described in Example 1 disclosed in the European Patent Application Open to Public Inspection No. 374837. "Blank" in Table 1 shows the values in the case where a high boiling point organic solvent was used. The condition of Blank is conventional as described in the official gazette of Japanese Patent Publication No. 56010/1986.

According to Table 1, the particle size could be controlled by the circumferential dispersing speed and stirring time. In the comparative example, the particle size could not be controlled, and the coloring density was only 103%.

[EXAMPLE 2]

The first solution of the following composition was obtained being stirred for one hour at 60 ° C.

Coupler (2)	5 kg
Ethylacetate	10 l

The composition of the second solution was as follows, wherein the dissolving conditions were the same.

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Water	50 l
Surface active agent (3)	2 l (Concentration 33%)

5 A dispersing operation was conducted in the same manner as that of Example 1 to remove ethylacetate. The particle size of dispersion and coloring density are shown in Table 2. It was ensured that the particle size could be controlled in the case of coupler (2).

TABLE 2

10

	20 m/sec 20 min	50 m/sec 20 min	Blank
Particle size	254 nm	105 nm	200 nm
Dmax	115%	104%	100%
MTF	0.68	0.70	0.57

15

20 [Spectral sensitization and coating recipe]

Silver iodobromide emulsion 0.9 g
 Spectral sensitizer (S-1) 0.0001 (mol/silver 1 mol)
 Spectral sensitizer (S-2) 0.0004 (mol/silver 1 mol)
 Coupler dispersing solution 0.5 (mol/silver 1 mol)
 25 Gelatin 1.0 g
 Coating aid (Su-1) 1.2 ml/200 ml of the above solution
 Thickening agent (V-1) 4% solution Controlled to be 32 cp
 Silver iodobromide emulsion: Silver iodobromide emulsion, containing the average grain size of
 30 0.7 μ m, was prepared by the double jet method, and the average iodo content of which was 7.5 mol%, was chemically sensitized to be the most appropriate sensitivity with sodium thiosulfate and chloroauric acid.

35 The coated sample was exposed to white light through a step wedge for sensitometry, and processed under the following conditions.

〈PROCESS〉

TABLE 3

40

Process	Processing Time	Processing Temperature	Replenishing Amount
Color Development	3 min 15 sec	38 $\pm$ 0.3° C	780 ml
45 Bleach	45 sec	38 $\pm$ 2.0° C	150 ml
Fixing	1 min 30 sec	38 $\pm$ 2.0° C	830 ml
Stabilization	60 sec	38 $\pm$ 5.0° C	830 ml
50 Drying	1 min	55 $\pm$ 5.0° C	-

In the above table, the amount of replenishment is expressed by the unit of ml/m² of photosensitive material.

55 〈Color Developing Solution〉

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	Water	800 ml
	Potassium carbonate	30 g
5	Sodium hydrogen carbonate	2.5 g
	Potassium sulfite	3.0 g
10	Sodium bromide	1.3 g
	Potassium iodide	1.2 mg
	Hydroxyl amine sulfate	2.5 g
15	Sodium chloride	0.6 mg
	4-amino-3-methyl-N-ethyl-N- (β -Hydroxylethyl)	
20	aniline sodium chloride	4.5 g
	Diethylene triamine pentaacetic acid	3.0 g
25	Potassium hydroxide	1.2 g

Water is added to make 1 l, and pH is adjusted to 10.06 using potassium hydroxide or 20% sulphuric acid.

(Color Developer Replenishing Solution)

35	Water	800 ml
	Potassium carbonate	35 g
	Sodium bicarbonate	3 g
40	Potassium Sulfite	5 g
	Sodium bromide	0.4 g
45	Hydroxylamine sulfate	3.1 g
	4-amino-3-methyl-N-ethyl-N- (β -Hydroxylethyl)	
	aniline sulfate	6.3 g
50	Potassium hydroxide	2 g
	Diethylenetriamine pentaacetic acid	3.0 g

Water is added to make 1 l, and pH is adjusted to 10.18 using potassium hydroxide or 20% sulphuric acid. (Bleach)

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Water	700 ml
1. 3 diaminopropane-tetraacetic acid ammonium salt	125 g
Ethylene diamine tetraacetic acid	2 g
Sodium nitrate	40 g
Ammonium bromide	150 g
Glacial acetic acid	40 g

5

Water is added to make 1 l, and pH is adjusted to 4.4 using aqueous ammonia solution or glacial acetic acid.

10

⟨Bleach Replenishing Solution⟩

Water	700 ml
1. 3 diaminopropane--tetraacetic acid ammonium salt	175 g
Ethylene diamine tetraacetic acid	2 g
Thorium nitrate	50 g
Ammonium bromide	200 g
Glacial acetic acid	50 g

15

20

Water is added to make 1 l, and pH is adjusted to 4.0 using aqueous ammonia solution or glacial acetic acid.

25

⟨Fixing Solution⟩

Water	800 ml
Thiocyanic acid ammonium	120 g
Thioammonium sulfate	150 g
Sodium sulfite	15 g
Ethylene diamine tetraacetic acid	2 g

30

35

Water is added to make 1 l, and pH is adjusted to 6.2 using aqueous ammonia solution or glacial acetic acid.

⟨Fixer Replenishing Solution⟩

40

Water	800 ml
Thiocyanic acid ammonium	150 g
Thioammonium sulfate	180 g
Sodium sulfite	20 g
Ethylene diamine tetraacetic acid	2 g

45

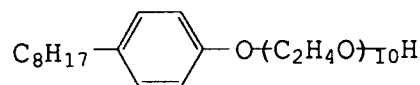
50 ⟨Stabilizing Solution and Replenisher⟩

55

Water	900 ml
The following compound A	2.0 g
Dimethylolurea	0.5 g
Hexamethylenetetramine	0.2 g
1, 2-benzisothiazoline-3-one	0.1 g
Siloxane	0.1 g
Aqueous ammonia solution	0.5 ml

Water is added to make 1 l, and pH is adjusted to 8.5 using aqueous ammonia solution or 50% sulphuric acid.

Compound A



As described above, according to the present invention, a hydrophobic substance of fine particles can be obtained irrespective of the kind of the substance, as well as the deterioration of sharpness and dye bleeding caused when a high boiling point organic solvent remains in a coated film can be prevented.

Claims

1. A method of dispersing a hydrophobic substance for photographic use in water, comprising:
 - preparing a first solution essentially consisting of said hydrophobic substance and a low boiling point organic solvent which is insoluble in water and a second solution, wherein said second solution essentially consists of water and a surfactant, or of water, a surfactant and a binder which is soluble in water;
 - mixing said first solution and said second solution to form a mixture;
 - stirring said mixture under a reduced pressure condition to evaporate said organic solvent from said mixture, thereby obtaining said hydrophobic substance being dispersed in said water.
2. The method of claim 1, wherein said mixing is carried out by a rotating member having the circumferential speed of 10 to 100 m/sec to exert on said mixture.
3. The method of claim 1, wherein said stirring is started within 5 minutes after said mixture has been formed.
4. The method of claim 1, wherein said stirring is carried out until the content of said organic solvent in said mixture has been lowered than 1 wt%.
5. The method of claim 1, wherein said reduced pressure condition is of 100 Torr in vacuum.
6. The method of claim 1, wherein said stirring is carried out under the temperature of said mixture of 40 to 80 °C.
7. The method of claim 6, wherein said temperature is 50 to 70 °C.
8. The method of claim 1, wherein said first solution includes a high boiling point organic solvent by the content not larger than 5 wt%.

Fig. 1

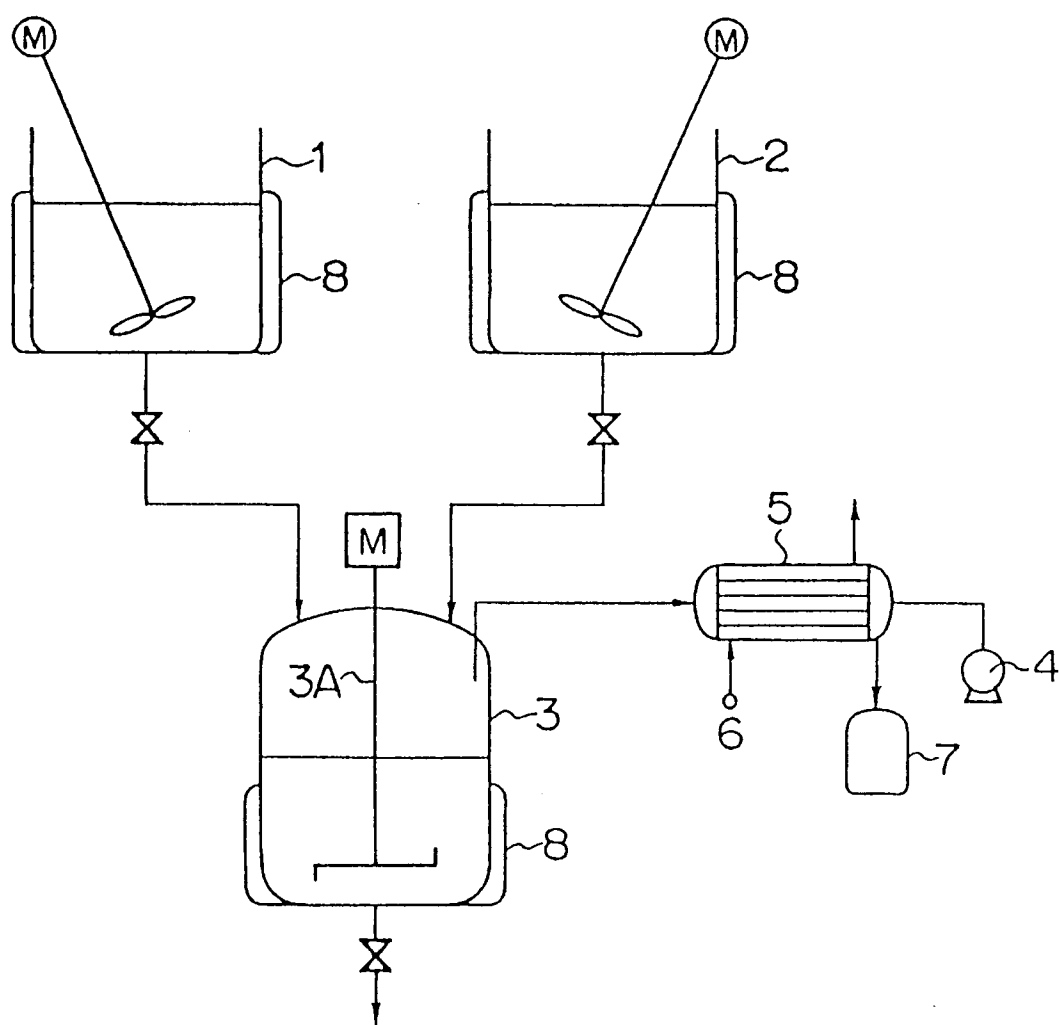
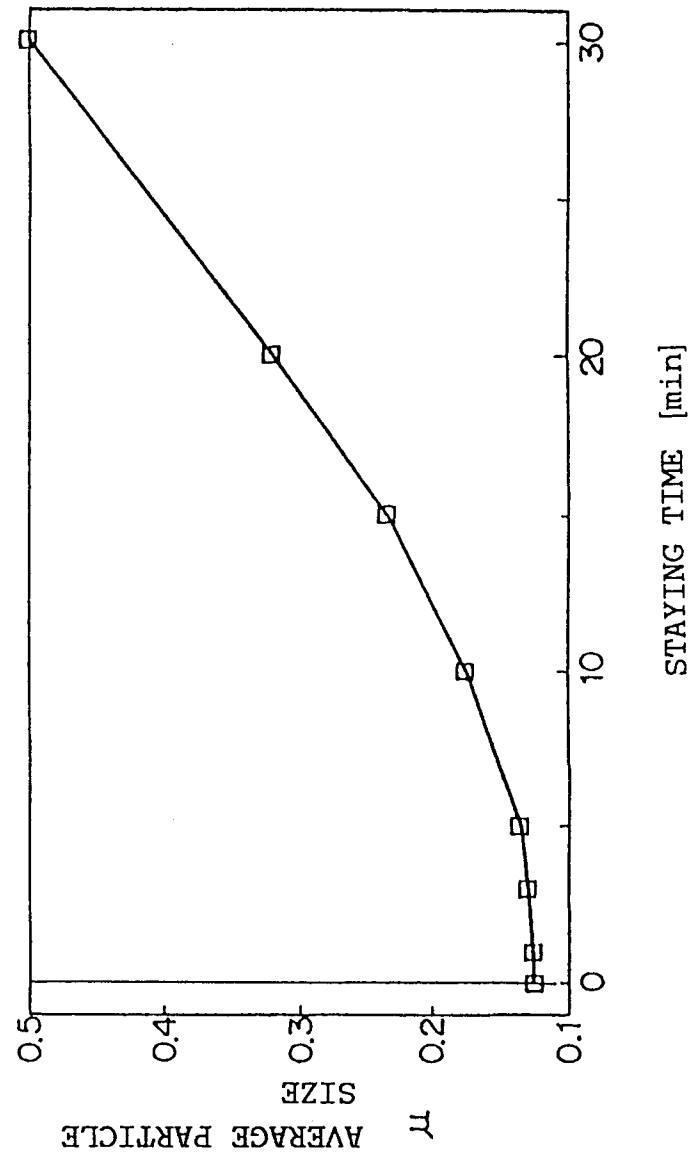


Fig. 2





European Patent
Office

EUROPEAN SEARCH REPORT

Application Number

EP 92 10 3789

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	GB-A-2 106 885 (FIJI) * abstract; figure * * page 1, line 50 - page 2, line 26 * * page 2, line 43 - line 47 * * page 3, line 65 - page 4, line 13 * * page 4, line 45 - page 5, line 38 * ---	1-8	G03C7/388 G03C1/005 B01F3/12
X	FR-A-2 101 616 (AGFA-GEVAERT) * page 8, line 20 - page 10, line 27 * * page 11, line 18 - page 14, line 11 * ---	1-8	
A	JP-A-53 112 731 (FIJI) * abstract * * page 2, left column, line 1 - line 19 * -----	1-8	
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			G03C B01F B29B
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 13 APRIL 1992	Examiner MAGRIZOS S.
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			